EFFECT OF SPAN LENGTH ON BENDING STRENGTH AND WEIBULL MODULUS OF REACTIVE ALUMINA BAR

A THESIS SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF BACHELOR OF TECHNOLOGY

by
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CERTIFICATE

This is to certify that the thesis entitled, **"Effect of span length on bending strength and Weibull modulus of sintered alumina bar"** submitted by MANOJ KUMAR MAJHI (107CR025) in partial fulfillments of requirements of the award of Bachelor of Technology degree in Ceramic Engineering at National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge the matter embodied in the thesis has not been submitted to any other university/institute for the award of any degree or diploma.

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Manoj Kumar Majhi
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ABSTRACT

The present study was undertaken to evaluate the bending strength of alumina both as a function of sintering temperature and span length. The fracture strength data was calculated on relatively a large number of samples for each data point (14). The strength data distribution was also used to evaluate the failure probability as well as the Weibull modulus which was correlated to the sintered density. It was observed that in the sintering temperature range of 1550 and 1600 ^{O}C the Weibull modulus showed that only one type of flaw behavior was operative in the sample and that the Weibull modulus increased with sintering temperature and decreased with increasing pan length. However at the highest sintering temperature 1650^{O}C the Weibull statistics couldn't be fitted to a single linear fit. This indicated that the data could be fitted to two different straight lines which implied that at 1650^{O}C, two different flaw types controlled the sample failure.

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CHAPTER 1

INTRODUCTION AND LITERATURE REVIEW

Single phase ceramics and ceramic –ceramic composites undergo brittle fracture due to the absence of required number of slip planes operating at room temperature. It is difficult to predict sudden and catastrophic fracture and failure of the components which are of brittle nature. This increases the risk of using these brittle materials (particularly ceramics) in strategic applications because their life time cannot be predicted with certainty. Moreover, in many ceramics, it has been observed that two samples prepared from same batch with identical processing may not fail at the same applied load. On the other hand, most of the ductile materials show identical failure behavior with almost all samples failing at or nearly same fracture stress. Further, in many ceramics and other brittle materials, surface conditions and irregularities, impurities etc (which may act as stress raiser) can significantly affect the failure behavior. In these disastrous failures the fracture behavior is understood by using fracture mechanics concept where the failure occur through unstable crack growth rather than through ductile manner [1-3]. The origin of crack extension was proposed by Griffith who postulated that crack extension in brittle materials occurs when there is sufficient elastic strain energy in the vicinity of a growing crack to form two new surfaces [4, 5] Later on, Irwin added the energy release concept to Griffith theory [6]. Irwin's approach helped to predict the strength behavior based on fracture toughness calculations (or in other words, resistance to crack propagation). The fracture toughness K_{Ic} concept relates the failure strength (σ_f) when the crack extends to the sizes of preexisting cracks or "flaws" (c) within a material.

$$\sigma_f = \frac{K_{IC}}{Y\sqrt{C}} \quad \text{……..(1)}$$

where *Y* is a dimensionless, material-independent constant, related to the flaw shape, location and stress configuration and is called the stress intensity shape factor "Flaws" in a ceramic material are generated from material Inhomogeneity, structural inhomogeneity, discontinuity

(like pores), pre-processing and post processing phase change, segregation etc [6]. These flaws are intrinsic or generated flaws and are distributed in some way throughout the material surface or volume. Griffith Equation says that materials having a smaller flaw size will have a higher strength and vice-versa.

The flaw size distribution in a material may have a normal or Gaussian distribution. (Fig.1). The failure will be initiated in the sample or material having the largest flaw within the highly stressed regions of the test specimen under tensile loading. In the event of a random sampling ,if a material contain large flaw , the strength distribution will be shifted towards the end of the tail at the right of the distribution (Fig. 1) and the material will have very low strength or fragile.

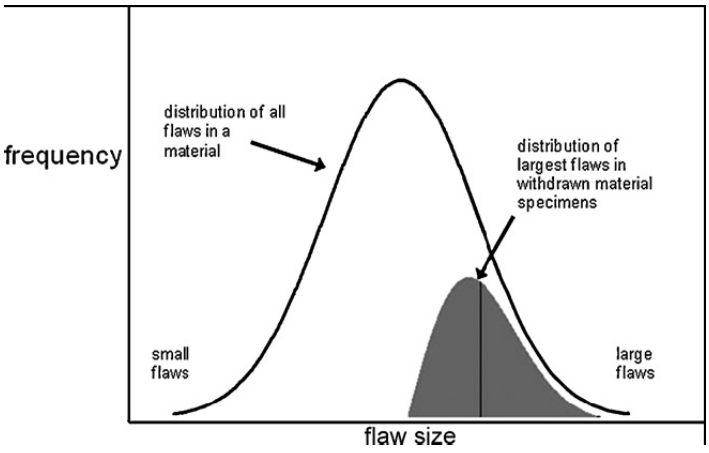

Fig. 1 – Total flaw distribution in a material (curved line).Withdrawing multiple test pieces from the total flaw population and collecting the largest flaw from each test piece results in a different distribution of "largest flaws"(shaded area) [8].

However, if the largest flaw in the test specimen is found towards the left side of the distribution (near the peak on its either side), the material will have relatively higher strength . The "largest

flaw" distribution might look like the shaded portion of Fig. 1 and the flaw distribution is usually not expected to be symmetric. Since a larger test specimen contains more flaws, it is more likely to contain a very large flaw (which may be strength limiting). For large sized specimens, the probability of finding a large (critical or strength limiting) flaw is more. This is distinctly different from smaller size samples, wherein the strength values are usually overestimated (due to less probability of finding a strength limiting flaw in a smaller area) [1, 8]. Thus an increase in sample size effectively shifts the "largest flaw" distributions towards the lower strength. The strength variation of a material having a distribution of strength limiting flaws is described by the goodness-of-fit of any of the extreme value distributions and it depends on the shape of the flaw distribution tail. In this regard, the Weibull distribution, is usually considered the best choice because the lowest possible fracture strength (σ_o) is zero in this distribution function, and the various parameters allow comparatively greater shape flexibility. Weibull distribution can provide reasonably good failure forecasts with small numbers of test specimens and it provides a simple and useful graphical plot [8]. In Weibull fracture strength analysis, the cumulative probability function, the probability of failure, (P_f), increases with the fracture stress variable

The threshold stress parameter σ_μ represents a minimum stress, below which test specimens will not break. The distribution shape parameter, *m*, is called Weibull modulus [9, 10]

Jayatilaka and Trustrum[11] have used fracture mechanics concept to develop a general expression for the failure probability using several general flaw size distributions. They correlated the experimentally obtained flaw size distribution with the fracture mechanics criterion. They have carried out analysis based on generalized flaw size distributions and have reached the same conclusions. Subsequent work, has also confirmed the power law function for the distribution of large crack sizes. Today Weibull statistics is routinely being used for characterizing failure of brittle materials. [13]

It is said that: “Ceramic strength data must meet stringent quality demands if they are to be used to determine the failure probability of a stressed component. Statistical fracture theory is based on the premise that specimen-to specimen variability of strength is an intrinsic property of the ceramic, reflecting its flaw population and not unassignable measurement errors. Ceramic strength data must be essentially free of experimental error” [14] . A blunt flaw, such as a circular or elliptical pore, is less critical than a sharp flaw, such as a micro crack. A material under load may break from a sharp flaw but may not break from a blunt flaw of a similar size. Depending on which type flaw control the fracture strength, the strength distribution will be different. However, if there is no particular type of strength limiting flaw then the material can show mixed type distribution. Bends or kinks in a Weibull distribution function are often indicative of fracture resulting from multiple flaw types. If the Weibull fit is not good (poor R^2 value), it suggests that the underlying flaws are inconsistent (wide distribution) and more than one flaw type or size can be strength limiting flaw. On the other hand, a good Weibull fit indicate that strength is controlled by a single flaw type [15]. A very important factor in determining Weibull statistics is the requirement of large number of test specimens in order to the completely characterize the strength distribution. The optimal number of test specimens depends on many variables, including material and testing costs, a general rule-of-thumb is that approximately 30 test specimens should be tested for obtaining a valid parameter [16]. The Weibull statistics can be used to predict changes in distributions according to the physical size of the individual test specimens.

CHAPTER 2

Objective

As elaborated in the introduction and literature section, the mechanical properties of brittle materials is a matter of utmost concern and therefore extreme care should be taken in studying, analyzing and reporting of strength data. Moreover, the strength values are also dependent on the testing method and sample size. In case of bending strength measurement (which is widely used for determining the fracture strength if ceramics), the strength also depends on span length . the study was undertaken to study the effect of sintering temperature (which will affect the sintered density and hence the residual pores, voids or flaws)as well as span length on the strength and Weibull modulus of Alumina.

CHAPTER 3

Experimental Work

3.1 Binder Mixing and preparation of powder for pellet making:-

Reactive alumina powder from Alcoa was used. A weighed amount of reactive alumina powder was taken and mixed with PVA (4%) binder .It was then mixed thoroughly and was scrapped out with the help of a spatula. After that, it was put inside a dry oven for 24 hours to form a solid mass. After 24 hours, the dry product was taken out from the oven and ground to the fine particles of reactive alumina powder in a mortar. Now the powder was ready for die pressing.

3.2 Pressing of reactive alumina powder:-

For the preparation of reactive alumina bar,3 gm powder was put in a rectangular die size (60×5×5)high carbon high chrome die)such that distribution of powder was uniform in order to give a uniform density of the bar . The powder was pressed in hydraulic press under a load of 10 Tons in three cycles to make a bar of dimension (60×5×5) mm^3. Before using the die , it was properly cleaned using acetone. 3% Stearic acid solution in IPA was used as lubricant for smooth motion of punch and easy removal of the pressed product. All bars were made with same procedure described above. For the pressing of bar, 10 ton load was used in three segments.

Pressure cycle:-

Segment -1 hold for 30 second at 3 Ton load.

Segment -2 hold for 30 second at 6 Ton load.

Segment -3 hold for 60 second at 10 Ton load.

Then dimension of the green sample was taken using digital slide caliper.

3.3 Sintering of reactive alumina bar:-

The bar were sintered at 3 different temperature (1550^0C,1600^0C,1650^0C) for 2 hour and holding at 750^0C for 30 minute in each case for the binder burn out.

Firing cycle:-

Temperature was increased from 0 ^{0}C to 750 ^{0}C at a heating rate of 5^0C/minute and then, held at that temperature for 30 minute for binder burnout .The temperature was further increased from 750 ^{0}C to final sintering temperature with a rate of 2^0C/minute and it was held for 2 hours. Dimensions of the fired samples were also taken by using digital slide caliper.

3.4 Calculation of volume shrinkage:

Volume shrinkage was calculated by measuring the dimension change from in initial volume(volume before firing) to final volume(volume after firing) .

$$\text{Volume shrinkage} = \frac{\text{Initial vol.-Final vol.}}{\text{Initial vol.}} \times 100$$

3.5 Bulk density of reactive alumina bar:-

Densities of the sintered pellets were measured by using Archimedes principle. Kerosene was used as the medium for density measurement. Bulk density was calculated by using formula

$$\text{B.D} = \frac{D \times 0.8}{W - S}$$

Where,

D= dry weight

W=soaked weight,

S=suspended weight

0.8 is the sp. Gravity of the medium (kerosene)

Procedure:-

Step-1:-dry weight of the each bar was taken. Then avg. dry weight was calculated.

Step-2:-The weighed bars were put in kerosene to make the bar pore free by filling the pore with kerosene hence bar was free from air bubbles, then weight of the bar was taken by putting inside kerosene in suspended condition which is known as suspended weight. The avg. suspended weight was calculated.

Step-3:-After taking the suspended weight bar was taken out from the kerosene and soaked by tissue paper and then weight was taken which is known as soaked weight , avg. soaked weight was calculated. The bulk density was found out from the measured data.

3.6 Polishing and Grinding of Sintered Bars

The sintered bars were ground and polished with SiC slurry to remove the surface roughness and to make the surface smooth, flat, and parallel. The edges were chamfered by using SiC slurry to remove the sharp corners and edges which can increase the local stress concentration.

3.7 Bending strength of sintered alumina bar

The strength of the sintered samples was measured in a Universal Testing Machine (UTM, Model HK10S TINIUS OLSEN). The breaking load was obtained from machine. 3-point bending strengths of the bars were calculated from the following equation.

$$\text{Fracture Strength} = \text{MOR} = Mc/I = 3PL/2bd^2$$

Where,

L is the span length,

P is the breaking load.

b is width

d is the thickness of the bar.

3.8 Weibull Modulus:

The Weibull modulus It is based on the failure of weakest link .It gives the strength distribution in a ceramic material in the form of mathematical expression. A given volume of ceramic under a uniform stress will fail only at a severe flaw. Thus, it presents the data in a format of probability of failure P verses applied stress σ , where P is the function of stress and volume (V) or area(S) under stress& is given by

$$P = f(\sigma, V, S)$$

Weibull proposed the following relationship for ceramics:

$$P(\sigma) = (\sigma - \sigma_\mu)/\sigma_o)^m$$

Typical graph between failure probability vs σ was shown

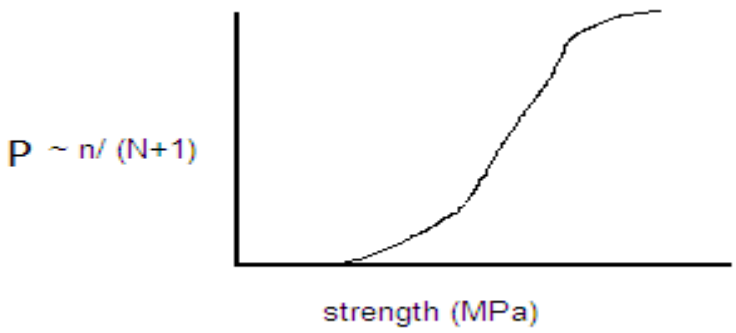

Fig 3.1 Failure probability vs strength

P is the failure probability = n/N+1

Where n is the rank of sample and N is the total number of samples .The weibull fracture strength analysis is a cumulative probability function in which the failure probability, P_f is found

$$P_f = 1 - \exp\left\{ - \left(\frac{\sigma - \sigma_u}{\sigma_o} \right)^m \right\} \quad \text{.....(1)}$$

Where σ_u is the threshold stress parameter and is defined as the minimum stress level below which no fracture will take place.

σ_o is the characteristic strength, which is dependent on the stress configuration and test specimen size.

M is the distribution parameter, known as Weibull modulus.

Equation (1) is known as three parameter Weibull function . However, for strength data evaluation only two parameter Weibull functions are used. The two parameter Weibull function is obtained from these parameter Weibull functions by setting $\sigma_u = 0$

$$P_f = 1 - \exp\left\{-\left(\frac{\sigma}{\sigma_o}\right)^m\right\}$$

…….2

Taking double logarithm of equation (2) yields

$\ln (1- P_f) = - ((\sigma/\sigma_o)^m)$

$\ln [\ln\{1/(1- P_f)\}] = m \ln\sigma - m \ln\sigma_o$

Thus a plot $\ln[\ln\{1/(1- P_f)\}]$ vs. $\ln\sigma$ would a straight line (or best fitted straight line) whose slope 'm' will be Weibull modulus. The slope (m) indicates the strength distribution width. A lower m value implies wide range of strength distribution which is primarily due to wide variation in flaw size.

The above equation hence becomes an equation of straight line with slope m

Thus the graph between $\ln [\ln(1/1-P_f)]$ vs $\ln\sigma$ is as follows

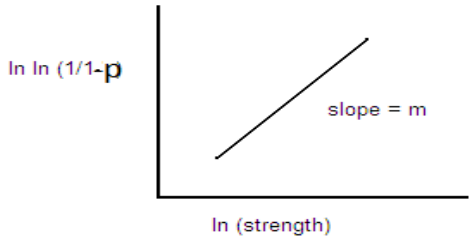

Fig 3.2 Plot for calculating Weibull modulus

A plot between failure probability (P_f) and strength (σ) is a S shape graph [Fig (3.2)]but this curve gives only an approximation of the probability of failure and does not yield the m value. However, when $\ln [\ln(1/1-P_f)]$ is plotted against $\ln\sigma$ it gives a straight line fit (or best fit) which provide a much closer insight about the failure probability. The Weibull curve is used extensively in depicting reliability or predicted reliability of materials or the components of the material.

Chapter 4

Result and discussion

4.1 Volume Shrinkage on Sintering

The volume shrinkage of the sintered pellets after sintering at different sintering temperatures are given in Table 4.1

Table – 4.1 Percent volume shrinkage for different sintering temperature.

No.	Sintering temp.(^{0}C)	Vol. shrinkage
1	1550	25.34%
2	1600	29.83%
3	1650	35.63%

.From the above table, it was inferred that with increase in sintering temperature shrinkage increased or in other words, the sample underwent better densification. During sintering process ,higher temperature causes enhanced mass transport which helps in pore removal .This hence forms more closed structure and hence volume decreases. Lowest volume shrinkage (25.34%) was obtained for the sample sintered at 1550^{o}C as the sintering temperature increased from 1550^{o}C to 1650^{o}C, the volume shrinkage increased due to increased densification and the highest volume shrinkage was obtained 35.63% in sample sintered at 1650^{o}C.

4.2 Bulk density:-

Table – 4.2 Bulk Density of Alumina for different sintering temperature

Sintering Temperature(^{0}C)	Bulk density(gm/cc)	Relative density
1550	3.7	0.92
1600	3.72	0.93
1650	3.78	0.94

The above Table shows that as sintering temperature increases from 1550^{o}C to 1650^{o} , relative density increases from 0.92 to 0.94. As temperature increases, sintering rate enhances due to change in diffusivity of diffusion species. Thus with the increase in sintering temperature, pore removal rate increases and particles come closer and thereby removing the pores and increases the sintered density of samples.

4.3 Bending strength as the function span length for different sintering temperature

Sintering temp.(^{o}C)	Span length(mm)	Avg. strength(MPa)
1550	20	317.3±38.1
	30	182.4±29.0
	40	93.6±20.7
1600	20	377.3±24.9
	30	192.8±13.3
	40	112.2±15.6
1650	20	396.8±30.96
	30	231.4±48.36
	40	190±20.2

The strength variation (Table – 4.2) of sintered alumina as a function of both span length and sintering temperature have also been plotted in Fig (4.1)

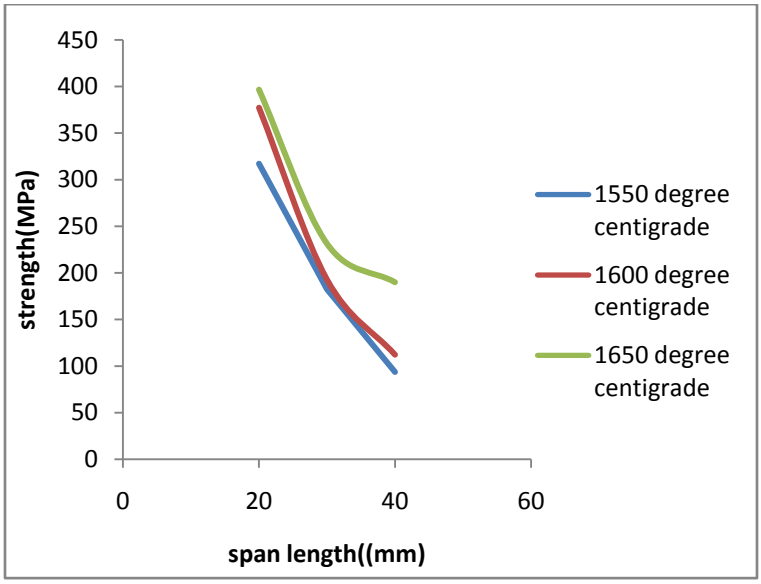

Fig4.1 Variation of Bending Strength as a function of Span Length and Sintering Temperature

Discussion

Table (4.3) shows the effect of varying span length on strength of samples sintered at different temperatures. Fig (4.1) shows that with increase in span length for samples sintered at a constant sintering temperature, the strength shows a decreasing trend because as the span length increases the area under load correspondingly increases. Hence, the probability of finding critical size flaw or strength limiting flaw being operative under maximum loading area increases at larger span length. Thus strength shifts to lower value.

Fig (4.1) also shows that for a fixed span length the sample strength increases with increase in sintering temperature. This behavior can be explained by consideration higher densification at higher sintering temperature which results in lesser number of large flaws in the sintered sample (thus the possibility of having a critical size flaw decreases). The fracture strength shifts towards higher load.

4.4 Probability of Failure as a Function of Sintering temperature and span length

The failure probability calculated using two parameter Weibull function is plotted against strength [Fig (4.2) to (4.4)]. The Figure represents the failure probability against strength for all the three different span lengths (20 mm, 30 mm and 40 mm) at a constant temperature (1550^{o}C). Similarly, figure (3.5) to (3.7) represents the Failure Probability for three different span lengths when the samples are sintered at 1600^{o}C. Finally Fig 3.8 to 3.10 represents the Failure Probability for three different span lengths for 1650^{o}C sintered samples

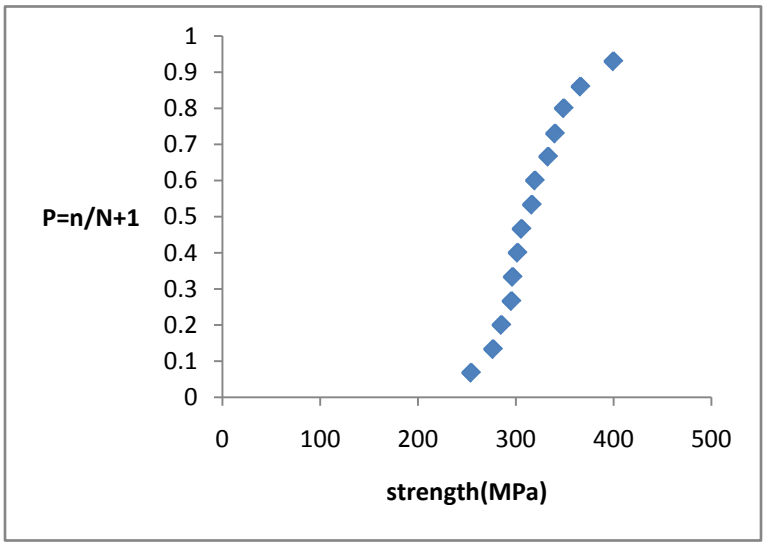

Fig 4.2 Probability of failure as a function of sample strength for sintering temperature 1550^{o}C (Span length 20 mm)

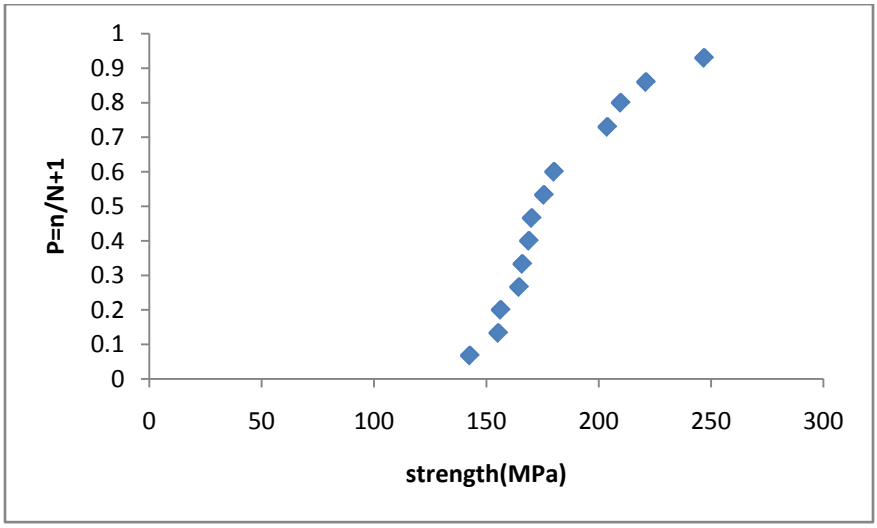

Fig 4.3 Probability of failure as a function of sample strength for sintering temperature 1550^{o}C (Span length 30 mm)

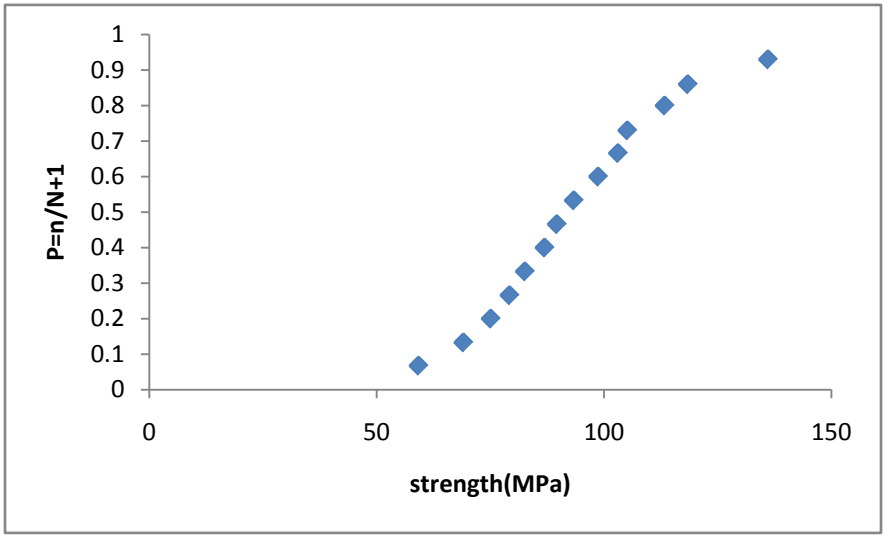

Fig. 4.4 Probability of failure as a function of sample strength for sintering temperature 1550^{o}C (Span length 40 mm)

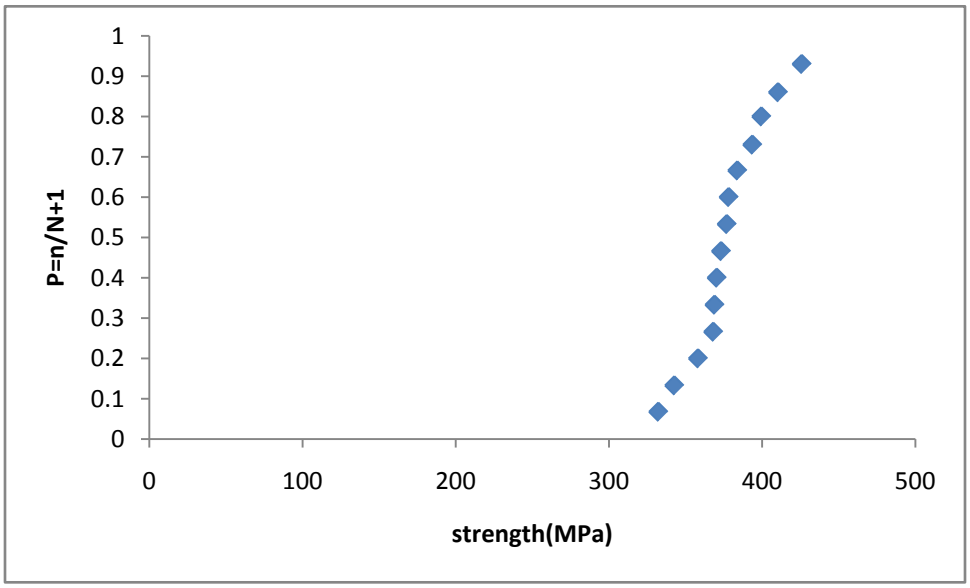

Fig.4.5 Probability of failure as a function of sample strength for sintering temperature 1600°C (Span length 20 mm)

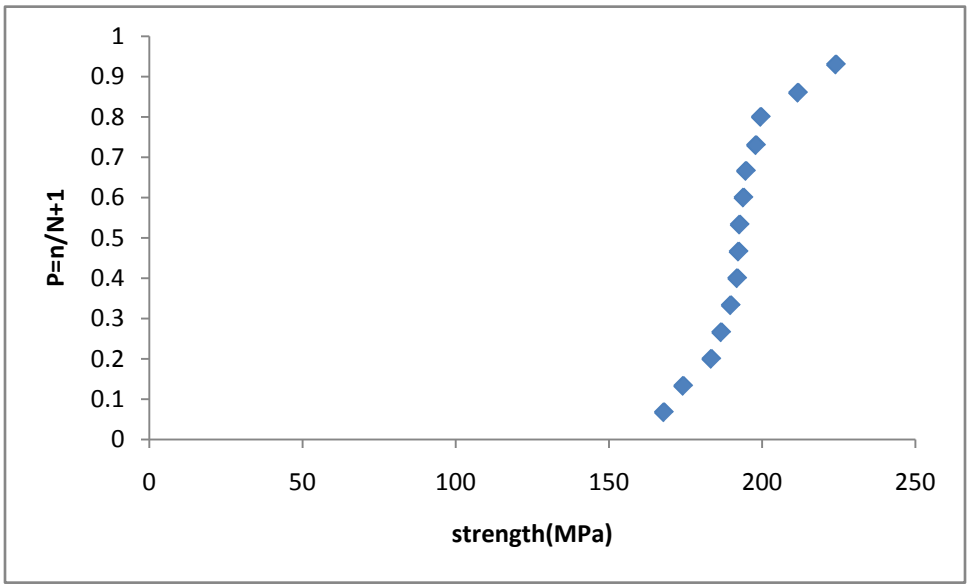

Fig.4.6 Probability of failure as a function of sample strength for sintering temperature 1600°C (Span length 30 mm)

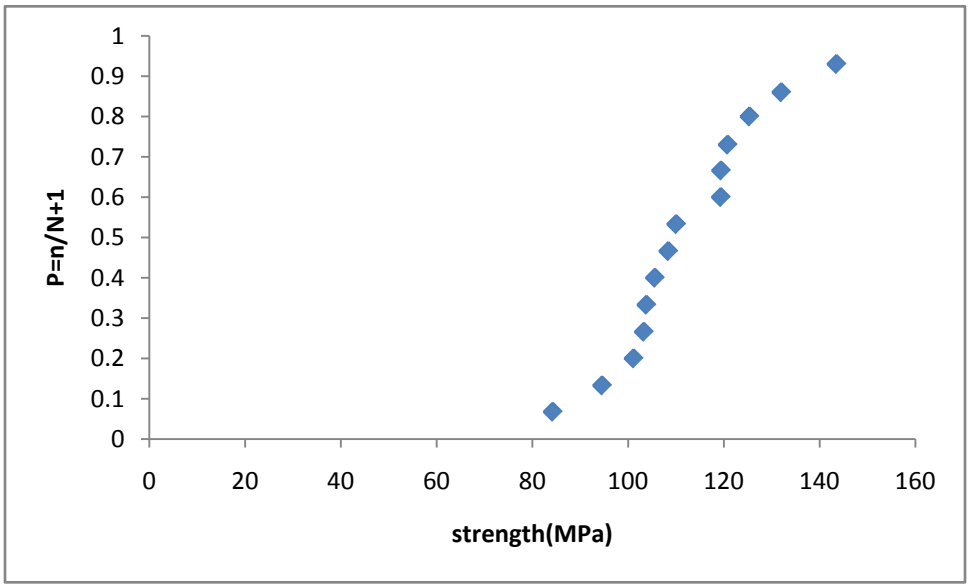

Fig. 4.7 Probability of failure as a function of sample strength for sintering temperature 1600^{o}C (Span length 40 mm)

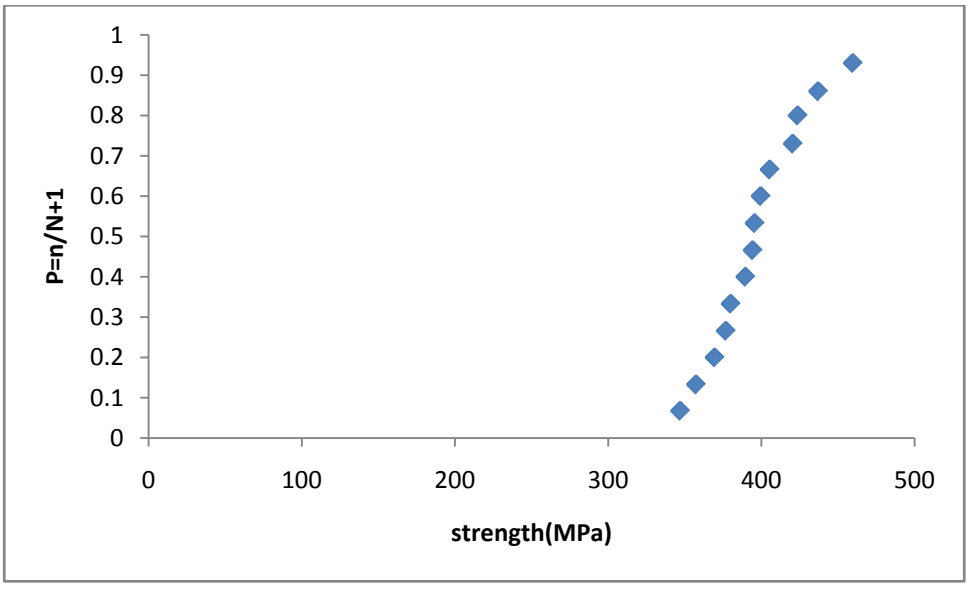

Fig.4.8 Probability of failure as a function of sample strength for sintering temperature 1650^{o}C (Span length 20 mm)

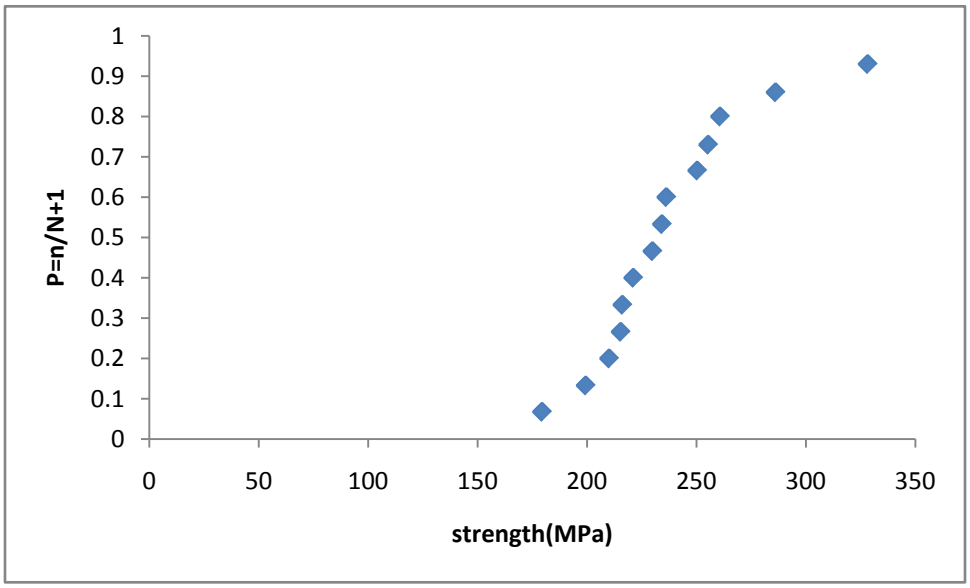

Fig.4.9 Probability of failure as a function of sample strength for sintering temperature 1650^{o}C (Span length 30 mm)

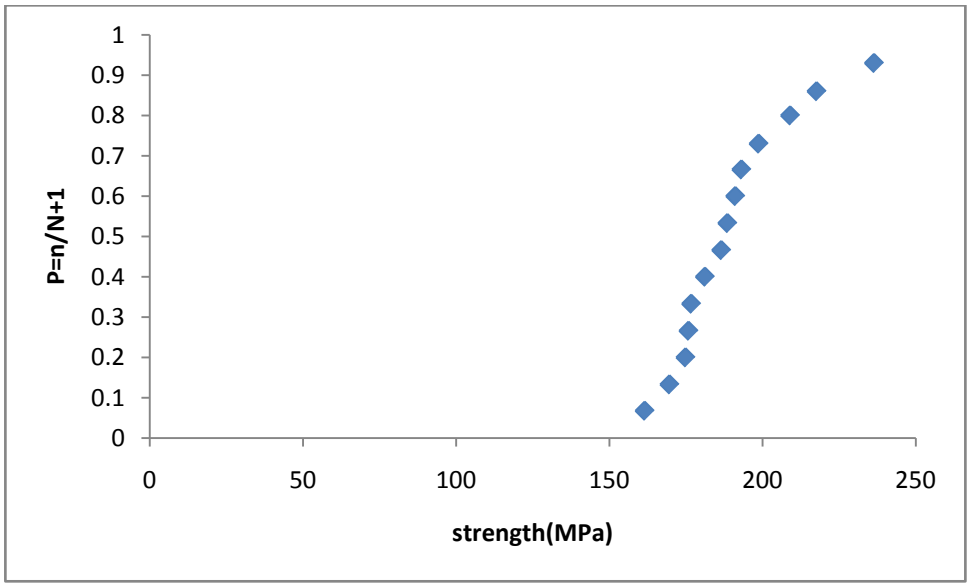

Fig. 4.10 Probability of failure as a function of sample strength for sintering temperature 1650^{o}C (Span length 40 mm)

All the above Failure Probability plots against fracture strength as shown above (Fig 4.2 to 4.10) show S-shape nature. At low load, the probability of failure is low. Failure probability increases as load increases because an increase in strength increases the crack propagation probability for cracks with smaller flaw size even. At very high load, the probability of failure is high and it remains unchanged with load because at this stage, even a very small flaw size can cause failure.

The Failure Probability curve also shifts towards higher load for samples sintered at higher temperature. This implies that at higher sintering temperature, the improved densification reduces the flaw size which makes the fracture difficult at lower load.

4.5 Weibull Modulus as a Function of Sintering Temperature and span length

The Weibull modulus is determined from Ln – Ln plot of failure probability against Ln strength. The plots are made for all the sample combinations – i.e. sintering temperature and span length combination. The results are shown in Fig 4.11 to 4.19

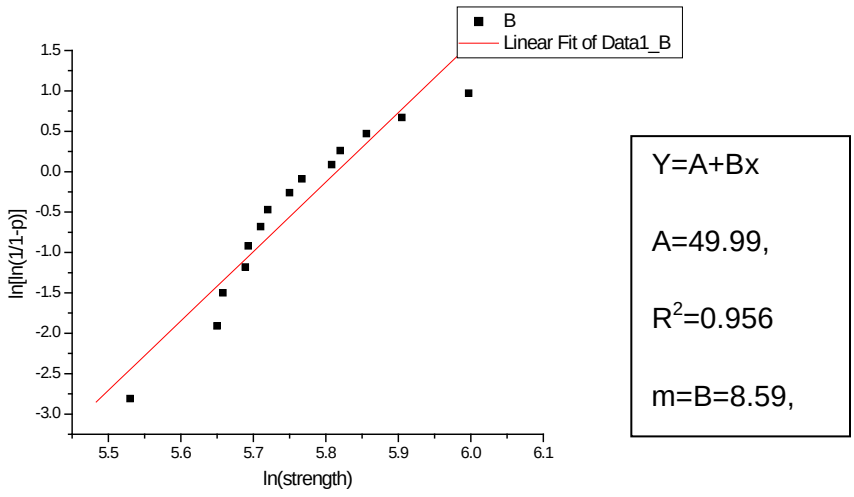

Fig.4.11 Weibull Modulus for Sample fired at 1550 and span length 20 mm

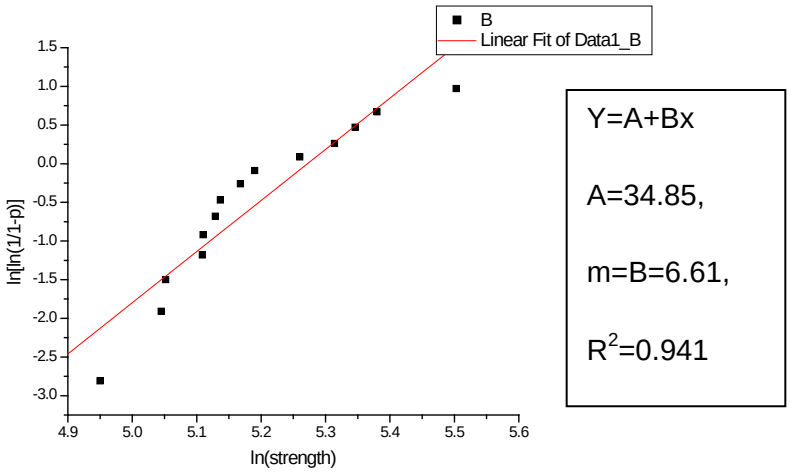

Fig.4.12 Weibull Modulus for Sample fired at 1550 and span length 30 mm

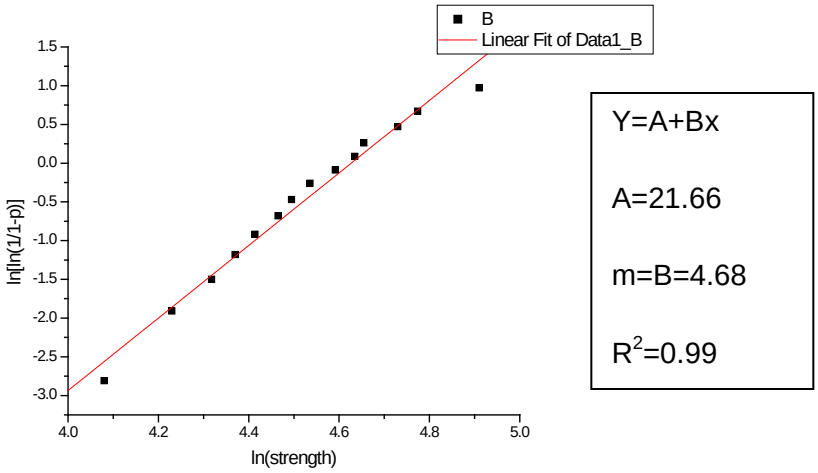

Fig.4.13 Weibull Modulus for Sample fired at 1550 and span length 40 mm

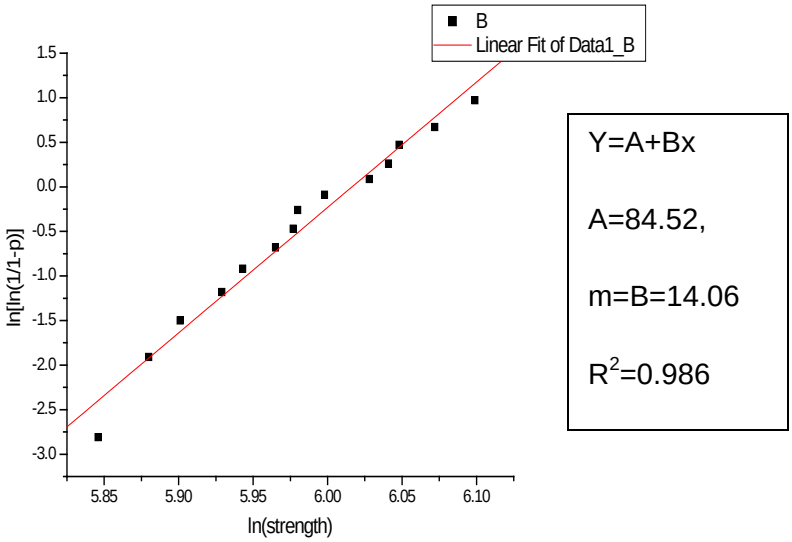

Fig.4.14 Weibull Modulus for Sample fired at 1600 and span length 20 mm

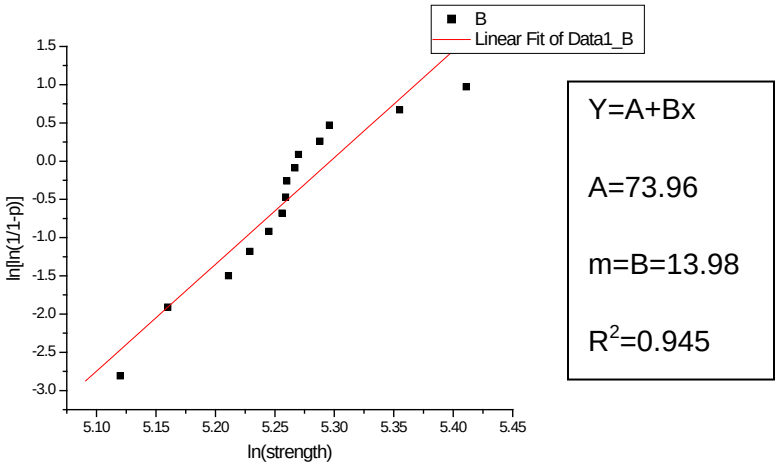

Fig.4.15 Weibull Modulus for Sample fired at 1600 and span length 30 mm

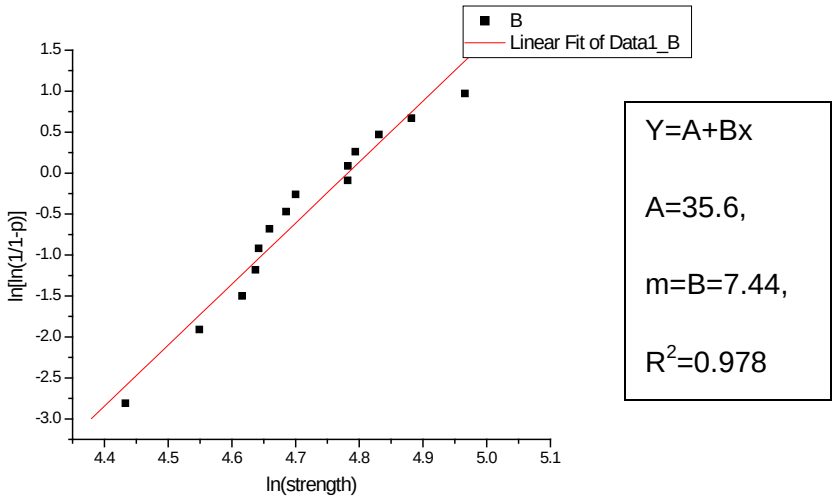

Fig.4.16 Weibull Modulus for Sample fired at 1600 and span length 40 mm

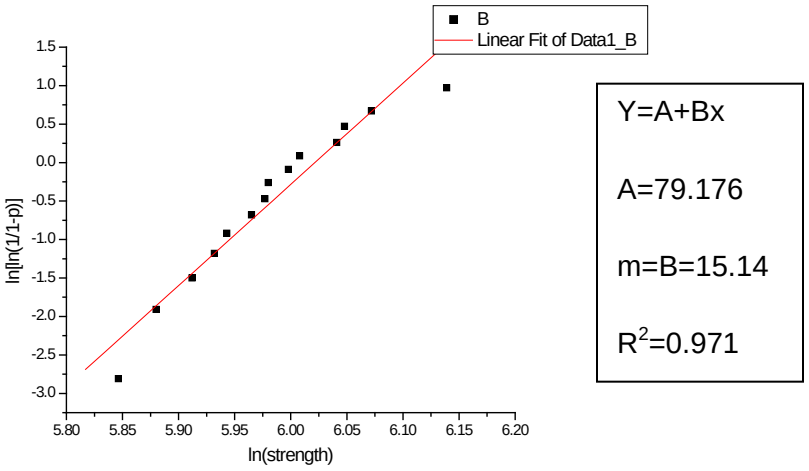

Fig.4.17 Weibull Modulus for Sample fired at 1650 and span length 20 mm

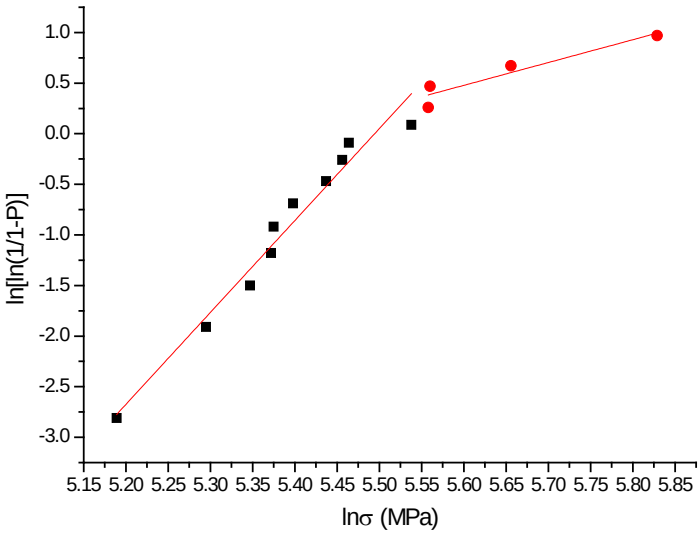

Fig.4.18 Weibull Modulus for Sample fired at 1650 and span length 30mm

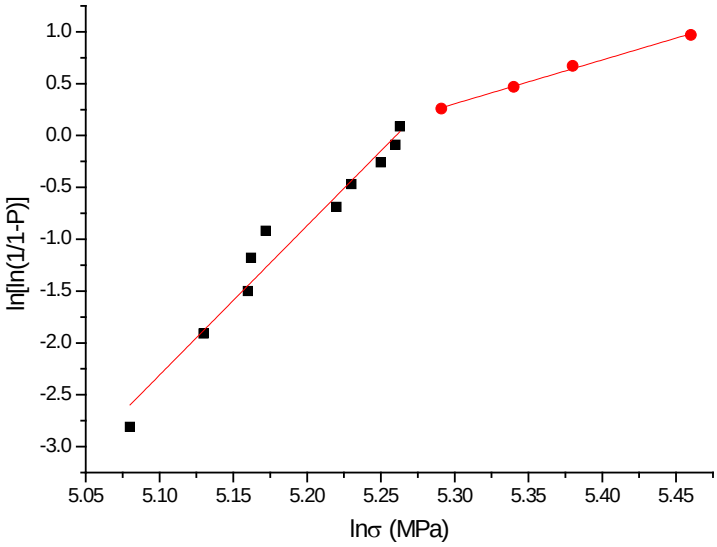

Fig. 4.19 Weibull Modulus for Sample fired at 1650 and span length 40 mm

Table 4.4 Weibull modulus (m) and survival coefficient (R^2) as a function of Sintering Temperature

Sintering temp.	Span length(mm)	Weibull modulus(m)	Survival coefficient (R^2)
1550^0C	20	8.59	0.956
	30	6.61	0.941
	40	4.683	0.99
1600^oC	20	14.06	0.986
	30	13.98	0.945
	40	7.44	0.978
1650^0C	20	15.14	0.971
	30	6.2	0.938
	40	9.66	0.943

Weibull modulus depends on many factors like particle size, particle size distribution, densification process (pressing of green sample and sintering temperature) and strength scattering factor .Higher is the sintering temperature, higher is the strength of the sinter product. Weibull modulus is directly proportional to the strength so, higher is the strength higher will be the Weibull modulus. Strength is inversely proportional to the span length hence, Weibull modulus is inversely proportional to the span length .So, from Table (4.4) Weibull modulus decreases as the span length increases for a particular temperature and Weibull modulus increases as the sintering temperature increases for a fixed span length .

Weibull modulus is inversely proportional to the scatter in the strength data i.e. if the strength value is highly scattered, probability of failure is more (according to the standard safety factor approach) and lesser will be the Weibull modulus which is also found in the present study [Table (4.4) for samples fired at 1600^0C]. However, for samples fired at 1650^0Cwhen tested in a span

length of 30and 40 mm, the data could not be fitted very well in single straight line fit. Two different fitting is necessary to fit the data. One type of fitting will be in the lower strength range and another at higher strength level. These type two step fitting has also been reported for many samples which implies that there is well defined flaw size distribution with at least two distinct type of flaw operating in two different stress level.

The other important parameter is R^2 value which is known as survival coefficient or co-relation coefficient .Survival coefficient can't be 1 for any grade of sample because 100% survival is impossible. Its value depends on the deviation of strength value from the best fit line which is clearly visible from the table data .Here, it is observed that highest value obtained was 0.99 & the lowest value obtained was 0.938. This implies that samples with a lower R^2value is more unreliable probably because of large flaw size and wide distribution of flaw size.

CHAPTER 5

CONCLUSIONS

- Sintered Alumina bar prepared from reactive alumina powders were sintered at 3 different temperatures (1550^{o}C, 1600oC, 1650^{o}C). The bulk density and volume shrinkage increases with increase in sintering temperature .with highest bulk density being 3.78 gm/cc (95% Relative Density) whiles the lowest being 3.71gm/cc (93% Relative Density). Highest volume shrinkage was 35% at sintering temperature 1650^{o}C and lowest volume shrinkage was 25% at the sintering temperature 1550^{o}C. The sintered alumina bar were tested for its flexural strength in 3 point bending using 3 different span length (20,30,40)mm, corresponding to each sintering temperature.

- It was observed that for a particular sintering temperature, bending strength decreased as span length increased but strength increased as sintering temperature increased. For a fixed span length, the .highest bending strength was 396.8 MPa for samples at 1650^{o}Cand tested at span length 20 mm and lowest bending strength found was 317.3MPa sintered at 1550^{o}C and tested at span length 20 mm. The measured strength values were correlated with Weibull modulus obtained by plotting of Ln[ln(1/1-P)] vs lnσ ,

- The Weibull modulus increased with increase in sintering temperature for a fixed span length and Weibull modulus decreased on increasing the span length for a particular sintering temperature The highest Weibull modulus was 15.14 sample sintered at 1650^{o}C and tested at span length 20 mm) and lowest one was 6.2 (sample sintered at 1650^{o}C and tested at span length 30 mm). Highest R^2value for Weibull function fitting was 0.99 and the lowest one was 0.938.

CHAPTER 6

SCOPE FOR FUTURE WORK

In future, the following work may be carried out in this area:

- The number of samples should be increased to at least 30 for data reproducibility
- Both three and four point bending strength data should be obtained.
- Detailed micro structural analysis (particularly fractography) need to be carried out.
- Grain size effect on the strength of sintered samples should be studied.

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